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DIAGENETIC BEHAVIOUR OF IRON SULPHIDES IN THE UPPER CRETACEOUS BLACK SHALES RED SEA COASTAL ZONE, EGYPT

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THE UPPER Cretaceous black shales of the Duwi Formation contain calcareous foraminiferal tests. phosphatized skeletal nektonic debris, fungal and algal forms as well as a noticeable bituminous matter. Also, a considerable amount of iron sulphides (pyrite and marcasite) are present.

The geometric distribution patterns of the iron sulphides revealed a high degree of congruency with the primary sedimentary fabrics of the shale and a close relation with the micro-organic strurctures of the bioclasts. These iron sulphides seem to develop through three successive diagenetic stages by segregation and filling processes. Framboidal pyrite spherules and pyrite crystal aggregates or clusters represent the early diagenetic stage (shallow burial to early cementation stage) followed by the formation of rod-shaped marcasite crystals, while authigenic coarse grained pyrite displays the intermediate to late diagenetic stage. Consequently, these sulphide forms are suggested to be a proper clue to the diagenetic history of these shales.

Morphologically, the framboids exhibit different shapes and types of ordering which are commonly obliterated due to the infilling process of additional pyrite.

Biogenic and physico-chemical conditions are assumed to control the diagenetic differentiations of the different generations of the iron sulphides, which might have taken place in a deep and calm marine environment.

INTRODUCTION

The Upper Cretaceous black shales of the Duwi Formation of the Red Sea coast, contain a considerable amount of iron sulphide developing in different forms. An attempt is made in this work to understand the genetic relation between the iron sulphide and the enclosing shale. Samples were collected from four phosphate mines, located in the area between Hamrawein and Quseir (Fig. 1). Oriented thin and polished sections cutting perpendicular to the bedding of the shales are systematically investigated. The obtained observations are based on the study of the geometry of the different iron sulphide forms and the country rock, which is the essential and inductive trend of modern genetic studies of rocks and ores.

According to : 1) the geometric fabrics exhibited by the iron sulphide and their relation to the primary sedimentary and organic structures of the shale and, 2) the geometric interrelationship between the developed iron sulphide forms themselves, the mode of formation and their paragenetic sequence were deduced. It becomes obvious that the occurrence of these iron sulphide forms could be regarded as an integral element in the diagenetic evolution of the studied black shales.

Particular attention was paid to the microscopy of the pyrite framboids which are present in predominant frequencies. The framboids are described and classified according to their shape, size, ordering and obliteration. The genetic event of the present framboids are also discussed.

The Upper Cretaceous black shales constitute a major part of the Duwi Formation (Fig. 1), and they are recorded to be of Campanian - L. Maastrichtian age (Said, 1962; Abd El-Razik, 1968 and Barakat and El-Dawoody, 1973). The sedimentologic and economic aspects of the black shales and the phosphorites of the Duwi Formation were treated in many works such as those by Mustafa and Ghaly, 1964; El-Tarabili, 1969; Soliman and Amer, 1972; Malak *et al.*, 1977; Glenn and Mansour, 1979; and Darwish, 1984.

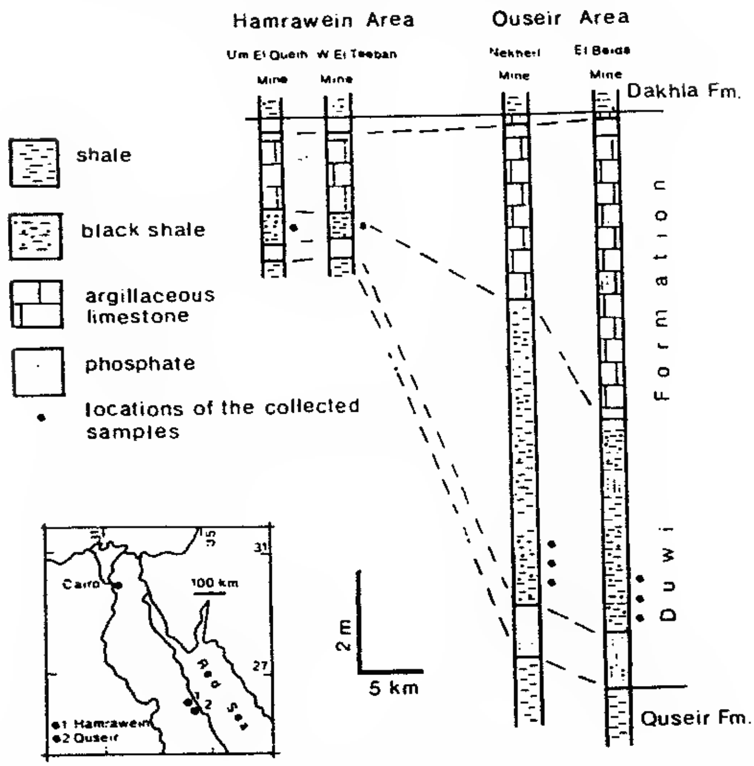

Figure 1. Location map and simplified stratigraphic sections of the studied black shales.

GENERAL PETROGRAPHY AND MICROFACIES

Petrographically, the studied black shales of the Duwi Formation show a general microfacies characterized by highly fossiliferous, bituminous, calcareous and partially phosphatized shale. The petrographic analysis and frequency of the components are shown in Table 1.

The argillaceous materials are generally arranged in a parallel manner giving rise to a characteristic fissility and laminae between which some elongated interlayered spaces were produced. Convolute and slump structures are predominantly developed, which may be accompanied by the gravitational deformational processes during the dropping of the bioclasts. Diagenetic small fractures or veinlets usually filled by cryptocrystalline quartz are also present. The clay minerals consist mainly of kaolinite, montmorillonite "smectite", chlorite and illite (Malak *et al.*, 1977). The lime mud fills some interclay spaces in random distribution.

Table 1. Petrographic analysis and frequency of components of the black shales of Duwi Formation. Terms used follow Folk (1968).

Fragmental Components						Orthochemical Components					
Bioclasts							Cement				
planktonic tests	nektonic remains	benthonic tests	algal & fungal debris	ferrugenous quartz grains	clay matrix	calcareous matrix "lime mud"	pyrite and marcasite	microsparite	silica	Organic components "bitumen"	Total
13	3	1	3	1	34	17	11	11	1	5	100

The bioclasts are represented mainly by complete planktonic foraminiferal calcareous tests with members of families *Globigerinidae* and *Globorotalidae* as well as some Rotalid forms (Fig. 3a). Very rare tests of *Bolimina sp.* and *Bigenerina sp.* are also present. It is noticeable that the benthonic forms are represented in very low frequency (not more than 5% of the total tests). The frequency of the nektonic animal remains commonly increases towards the underlying phosphorites. They are displayed mainly as fish remains (spines, scales, bones and fins), ranging from 100 to 1000 *u* in diameter. The nektonic debris are generally phosphatized consisting of fluorapatite and showing internal laminae structure (Fig. 4e), (Philobbos, personal communication). The algal and fungal remains are usually accumulated in branched or unbranched streaky forms between the argillaceous matrix and commonly associated with bitumen (Fig. 3b). Some of these organic remains show internal lamellar structures which may be preserved by iron sulphide.

The quartz grains fall generally in the silt size grade, ranging between 30 to 50 *u* in diameter. They are of angular and elongated shape and usually scattered randomly with their long axes parallel to the argillaceous laminae.

The authochtonous cement is represented by the pre-dominance of iron sulphides and calcite with rare amount of silica (cryptocrystalline quartz). In polished sections, the iron sulphides occur in three main forms; 1) framboidal pyrite with clusters of small pyrite crystals, 2) marcasite and, 3) coarse grained idiomorphic pyrite.

From the described microlitho- and, microbio-facies associations, it is evident that these shales are deposited in outer neritic to marginal bathyal zone under deep and calm marine conditions.

GEOMETRIC DISTRIBUTION PATTERNS OF THE IRON SULPHIDE IN THE BLACK SHALES

The main microscopic geometric distribution patterns of the different iron sulphide forms are illustrated in Figure 2.

Group 1 displays stratiform or planar laminated patterns congruent with the primary bedding of the shale. Type Ia (Fig. 3c and 3d), forms streaks or fine laminae of framboids. In type Ib (Fig. 3b), the laminae are formed by separate small pyrite crystals. Type Ic represents streaky arrangement of framboids showing load structure (Fig. 3e). Type Id (Fig. 3f) shows convolute or slumping structure formed by pyrite framboids. Types Ie and If represent typical load and geopetal structures produced by buried fossils dropping into the framboid laminae of types Ia and Ic (Fig. 3g) or when iron sulphides fill the bottom chamberlets of the fossil tests (Fig. 3b).

In group 2 the iron sulphides occur mainly in the chamberlets of the microforminiferal tests. According to the forms of the iron sulphides occuring in the fossil chamberlets, group 2 is subdivided into the following types : i) foraminiferal tests filled with small pyrite crystals and pyrite framboids (type 2a in Fig. 2 and Fig. 3i); ii) tests filled with concentrically arranged small pyrite crystals of framboidal type (type 2b in Fig. 2 and Fig. 3g); iii) tests filled with small pyrite crystals and marcasite (type 2c in Fig. 2); iv) tests filled with pyrite framboids with marcasite (type 2d in Fig. 2); v) tests filled mainly with pyrite framboids (type 2e in Fig. 2 and Fig. 3k); vi) tests filled with small pyrite crystals, marcasite and framboids (type 2f in Fig. 2 and Fig. 3e) and vii) fossil chamberlets filled with combination of small pyrite crystals, marcasite and pyrite framboids together with coarse grained, idiomorphic pyrite (type 2g in Fig. 2 and Fig. 3 m). In some cases, the walls of the filled fossils of types 2e and 2g are replaced by marcasite. In the aforementioned types, the spaces between the iron sulphides in the fossil chamberlets are either filled by microsparite with or without cryptocrystalline quartz or being empty, forming primary vugs.

In group 3 (types 3 a-d), the iron sulphides are confined to certain crumpled or folded structures which were likely produced during deformational and compaction processes. Type 3a represents iron sulphides filling fossil tests and occurring within lateral crumpled streaks drawn away from the fossils (Fig. 4a). In type 3b, the iron sulphides occur only in the drawn out streaks of the fossils. Type 3c corresponds to isolated nebulite-like textures or geodesic forms consisting of microsparite with iron sulphides . Type 3d displays a highly

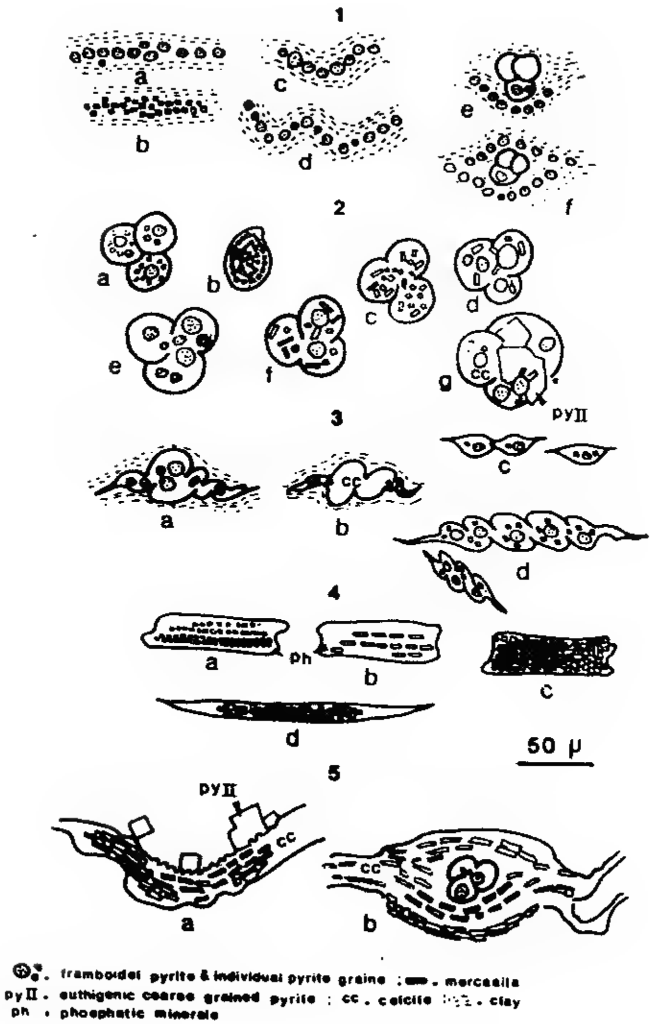

Figure 2. Geometric distribution patterns of the iron sulphides in the black shales (description in the text).

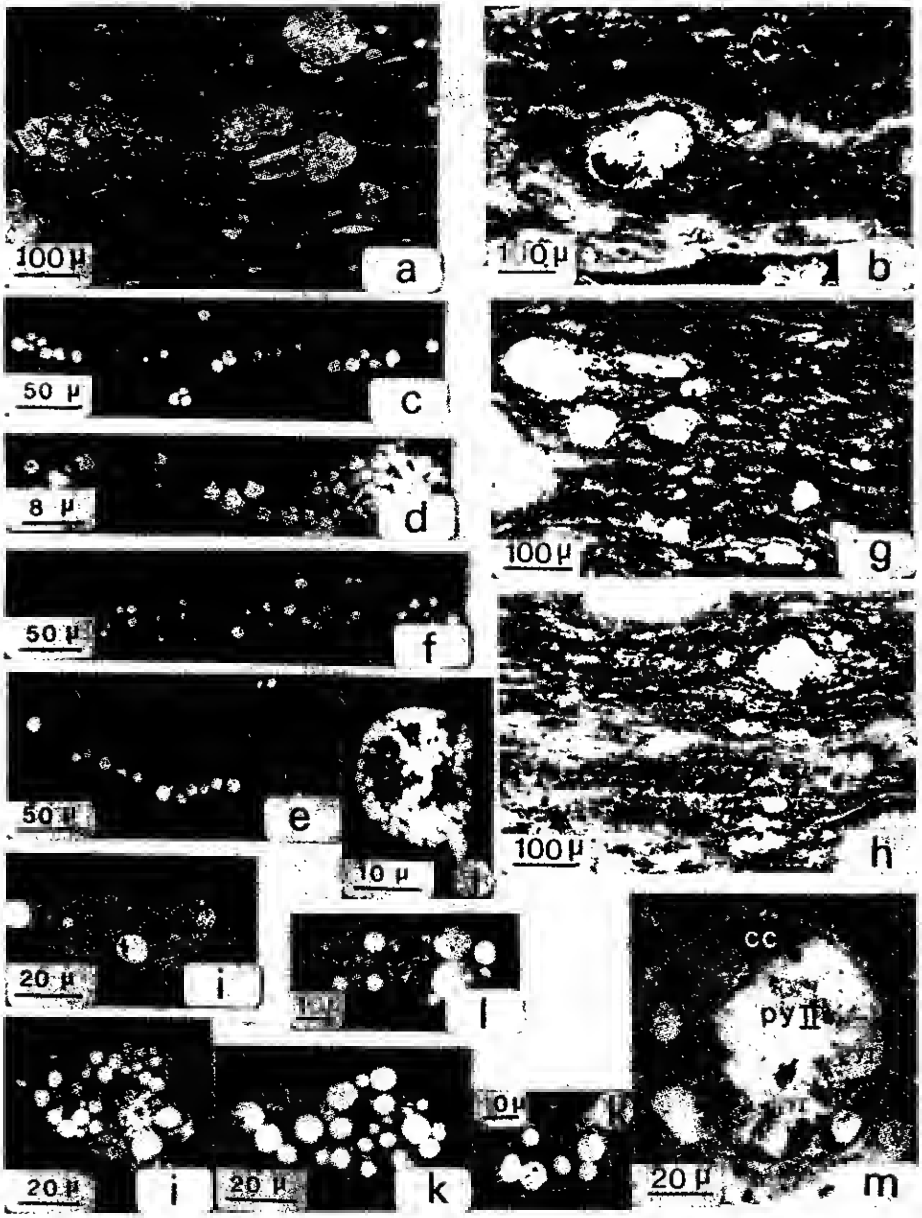

Figure 3. Photomicrographs showing the different forms of the iron sulphides in the black shales. (description in the text). py I = framboidal pyrite ; py II = coarse grained pyrite; mr = marcasite; cc = calcite.

crumpled and folded textures of microsparite and iron sulphides arranged in layered patterns or thrown discordantly within the clay laminae of the shale (Figs. 4b, c and d).

Group 4 represents iron sulphides localizing within vertebrate bone fragments (types 4a and 4b in Fig. 2 and Figs. 4e and 4f) or scales and fins of fishes (type 4d in Fig. 2 and Fig. 4c). The iron sulphides in this group are mainly represented by small pyrite crystals or rod-shaped marcasite. They are commonly arranged in distinctly parallel rows within the fragments, suggesting that they preserve the orientation of the micro-organic structures or fill oriented spaces between the tissues (Figs. 4g and 4h). Completely "pyritized" bone fragments are commonly observed (type 4c in Fig. 2).

Group 5 represents rod-shape marcasite crystals occurring within branched or unbranched filaments-like structrues (Fig. 5a), which are thought to be of biogenic origin (algal and fungal forms). The marcasite crystals are commonly arranged in undulated and parallel rows forming cell like structure or rarely form mosaic texture. Coarse grained idiomorphic pyrite grows usually on the marcasite (type 5a in Fig. 2 and Fig. 5b). Type 5b displays load texture and consists of buried "pyritized" fossils of types 2a and 2e dropping into the marcasite layers (Fig. 5c) and may also be covered with marcasite (Fig. 5d). Circular forms of marcasite or pyrite are usually trapped within the marcasite rows (Fig. 5d). Intergranular mosaic microsparite crystals commonly occurred within these biogenic forms filling spaces between the marcasite crystals.

Sedimentologically, the observed geometric patterns of the iron sulphides revealed a high degree of symmetry and congruency with the structures of the enclosing shales. They could be described as well-defined primary and deformational sedimentary fabrics. Moreover, these iron sulphides are closely related to the micro-organic structures of the bioclasts. From these observations, it could be concluded that the iron sulphides are formed during the diagenesis and before the complete consolidation of the enclosing rock.

DETAILED MICROSCOPIC DESCRIPTION OF THE DIFFERENT IRON SULPHIDE FORMS

1- Framboidal Pyrite and Small Pyrite Crystals.

The term "framboid" is used in the present work as defined by Rust (1935); i.e. "... a texture consisting of rounded aggregates of minute euhedral pyrite crystals massed together to give the appearance of raspberry ...". The framboids range in diameter from 1-100 u and they are common constituents of both ancient and modern sediments, from Precambrian to Recent (Love and Amstutz, 1966). They form an important part of the sulphide mineral content of

Figure 4. Photomicrographs showing the different forms of the iron sulphides in the black shales (description in the text).

many stratiform and stratabound sulphide deposits occurring in argillaceous clastic or carbonate sediments, e.g. Kupferschiefer, Rammelsberg, Mississippi Valley ... etc. The framboids are also present in : the Karst deposits (Zuffardi, 1976 and El Aref and Amstutz, 1983); the Recent beach sands (Papadakis and Michailides, 1977 and Papadakis and Amstutz, 1980; Recent lakes (Vallentyne, 1963a and 1963b); and in present-day formation of an exhalative sulphide deposit at Vulcano (Honnorez *et al.*, 1976). Pyrite framboids are also discovered in andesite by Love and Amstutz (1969). There are different theories dealing with the origin of the framboids as suggested by different authors. Schneiderhohn (1923) and Ramdohr (1953) believed that the framboids are the fossilized remains of sulphur bacteria; while Skripchenko (1970), considered them as metabolic products of globular bacteria. However, Love (1957); Love and Zimmerman (1961); Love and Murray (1963), suggested that the framboids represent fossil organisms. Schouten (1946), believed that the framboids are of inorganic origin, while Amstutz *et al.*, (1974), suggested a colloidal origin for the framboids.

The pyrite framboids, observed in the studied sections, are usually assciated with numerous aggregates of small individual pyrite crystals or clusters. The term "cluster" is used by Love and Amstutz (1966) for any aggregates of pyrite crystals that do not fit the definition of the framboids. The pyrite crystals are of square, six-sided or rectangular shapes in cross-sections. They range in diameter between 2 and 10 *u* with increasing abundance of the smaller crystals. They show signs of concentric arrangement or agglomeration which gradually form proper framboids (Figs. 5e, 5f and 5g).

The observed framboids occur mainly as isolated grains or groups. They range from 2 to 44 *u* in diameter. The framboids are of spherical and oval sectional shapes (Figs. 6 and 7). Some framboids exhibit forms of fossil chamberlets (Fig. 3j and Fig. 6j). The framboids are characterized by irregular or polygonal external outlines, which tend to be regular or smooth as a result of the infilling process described below.

From the wide variety of forms of the cross sectioned framboids illustrated in Figures 6 and 7, the pyrite crystals within the framboids show different grain sizes whatever the size of the spherules. They range in diameter between 2 and 8 *u* and exhibit different shapes of sections including: square, rectangular, hexagonal, rhombs, triangular, and trapezoid. These sectional shapes reflect the original pyrite forms to which the sections belong (Zimmerman and Amstutz, 1973). With respect to the internal geometric arrangement of the pyrite crystals within the framboids, two main groups have been recognized : "unordered" (Love and Amstutz, 1969) and ordered framboids. The former includes sections of pyrite which do not show any regularity of arrangement or predominant alignments (Figs. 6a-6d). The ordered framboids include pyrite crystals having a

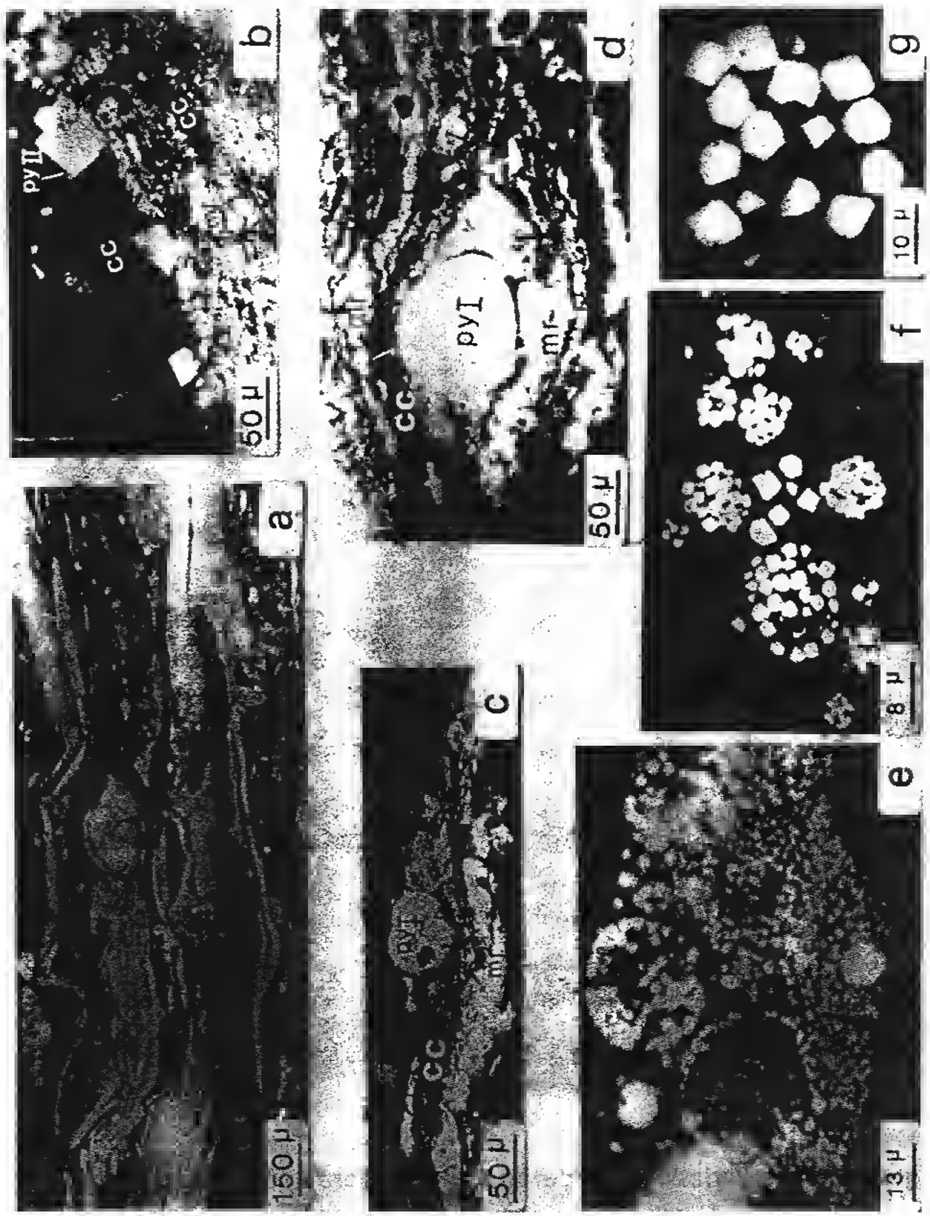

Figure 5. Photomicrographs showing the external and internal textures of the iron sulphides in the black shales (description in the text). py I = infilled framboids; py II = coarse grained idiomorphic pyrite; mr = marcasite; cc = calcite.

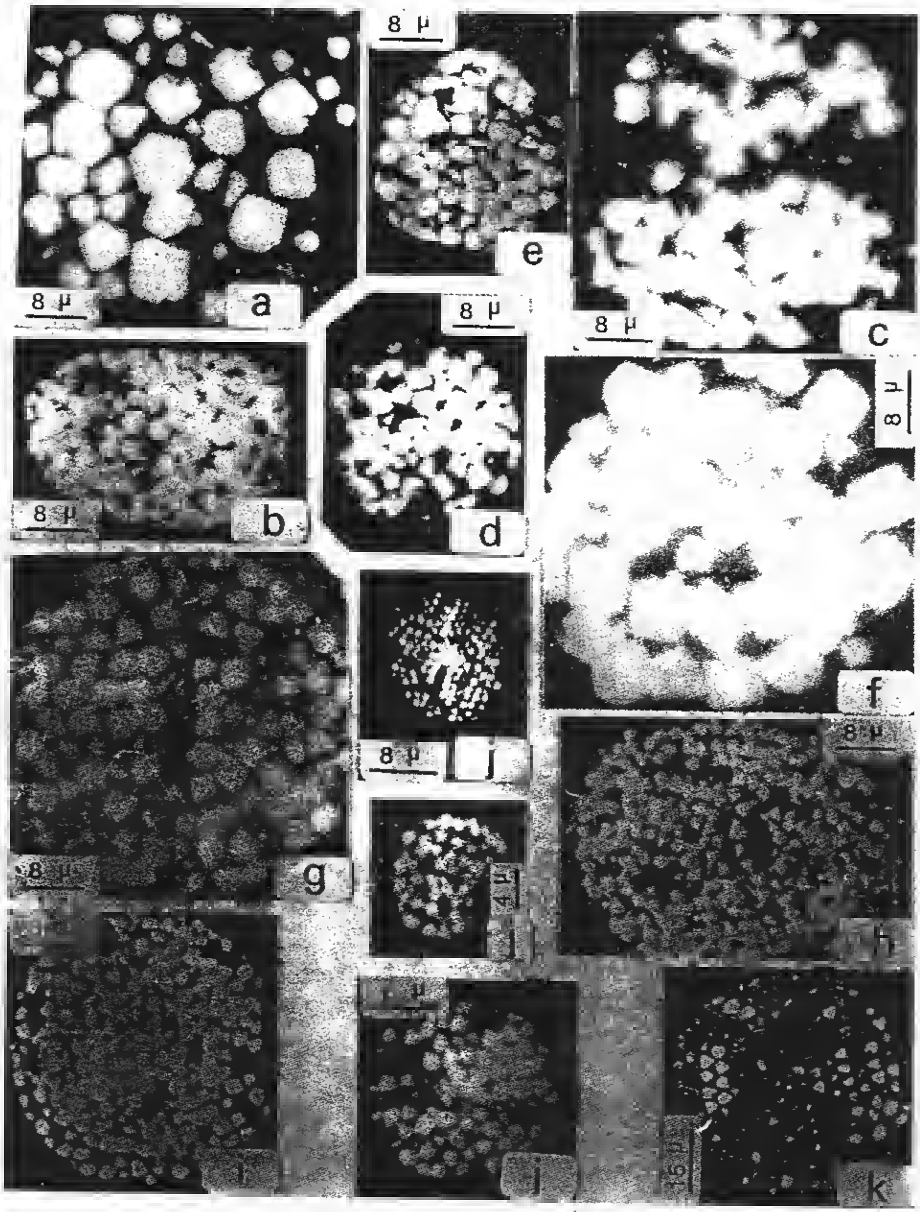

Figure 6. Wide varieties of ordered and unordered framboidal pyrite.

distinct shape and degree of adjustment. The crystals may be arranged in spiral forms (Figs. 6e and 6f) as a result of preservation of internal microfossil structure or they commonly form concentric rounded and(or) linear arrangement (Figs. 3g, 5f, 6g-6k and 7a). According to the shape of the pyrite crystals constituting the framboids, the ordered framboids could be classified into the following three main types: i) square type corresponding to type 1 of Love and Amstutz (1966) and represented by cubic crystals arranged essentially in cubic system of packing (Figs. 5f and 6g-6k), ii) triangular type consisting of traingular shapes perfectly arranged in concentric circular form (Fig. 6k). Such crystal shapes may be produced as a result of cutting of a closely packed cube or pyritohedron (Zimmermann and Amstutz, 1973); and iii) hexahedral type (Fig. 7a) consists of adjacent hexagonal crystals and corresponds to type 2 of Love and Amstutz (1966). This type could be obtained from cutting of closely packed layer of pyritohedra or cubes according to the determination of Zimmerman and Amstutz (*op. cit.*).

The internal structures of the studied framboids are commonly obliterated due to the addition of new pyrite or an "infilling" processes (Love and Amstutz, (1966). The infilling appears to start from the center of the pyrite spherules (Fig. 7b) and affect both the ordered and unordered framboids (Figs. 7c and 7d). By the advancement of the infilling process, the framboids become partially homogenous including small regular or irregular distributed holes (Figs. 7e and 7f). These holes may reflect the original internal structures of the framboids. An ultimate stage of infilling produce highly homogenized spherules characterized by smooth and regular external outlines (Fig. 7g). With regard to the size and the number relationships between the ordered and unordered framboids and the infilled spherules, 500 framboidal grains are measured by using wide field microphotographs and the obtained data were plotted in Figure 8. The ordered and unordered framoids represent about 30 % of the total and they lie in a unimodal distribution with maximum size around 3-5 *u*. The infilled framboids display 70 % of the total and are polymodal with peaks of 5, 7, 10 and 14 *u*. It could be concluded that the majority of the framboids have been affected by infilling process which do not depend on the size of the original ordered or unordered framboids.

The framboidal spherules are commonly joined together (Fig. 7h), forming groups of rounded forms or irregular masses (called super-framboids by Ncube *et al.*, 1978). The adjacent boundaries of the joined framboids are usually invisible due to the infilling effect (Fig. 7i).

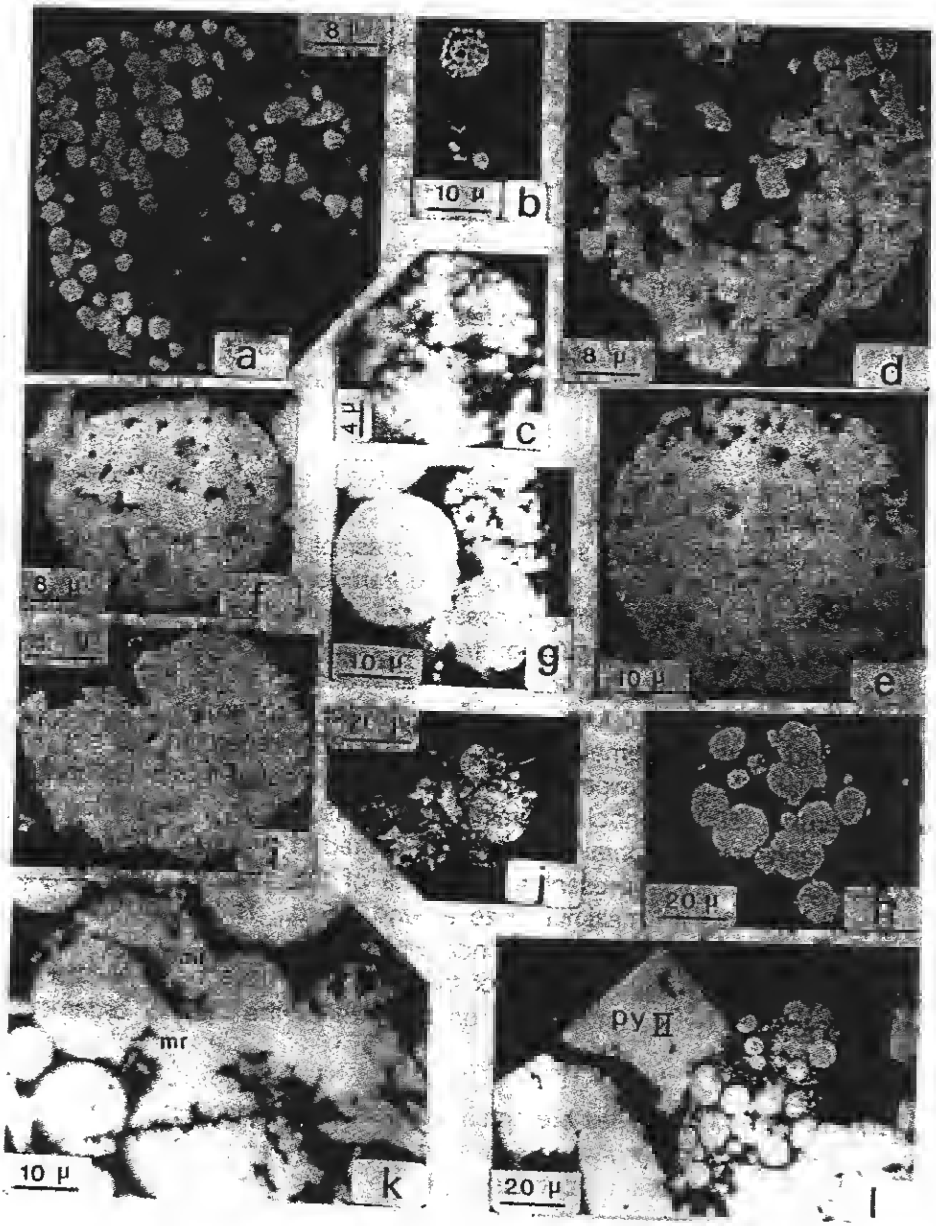

Figure 7. Wide varieties of ordered and infilled framoids. py II = coarse grained idiomorphic pyrite; mr = marcasite.

2- Marcasite

The marcasite occurs mainly as small aggregates of rod or cylinder shaped crystals, up to 10 *u* in length or rarely as mosaic grains. The marcasite crystals grow frequently above the outer surfaces of the framboids and fill spaces between them (Figs. 7i and 7k).

3- Authigenic Coarse Grained Pyrite

The pyrite of this form occurs as coarse grained crystals, up to 200 *u* in diameter. It shows well developed idiomorphic outlines in both polished and thin sections. The coarse grained pyrite grows authigenetically above the other described iron sulphide forms. It mostly shows convex surfaces against the framboids and the marcasite and is commonly overlain and cemented by microsparite with or without cryptocrystalline quartz (Figs. 5b and 7l).

DIAGENETIC CRYSTALLIZATION SEQUENCE

The studied iron sulphides form a major part of the constituting cementing materials of the shale. The geometric interrelation-ship of these minerals indicates that they are formed in three successive generations of crystallization or segregation processes corresponding to diagenetic crystallization differentiation or fractionation (Fontbote and Amstutz, 1982 and El Aref, 1984). The first crystallization generation is displayed by the formation of pyrite crystals (py I). This py I is followed by the crystallization of marcasite. Authigenic coarse grained pyrite (py II) displays the youngest iron sulphide generation in the crystallization sequence. With regard to the diagenetic history of the country rock, py I seems to have been formed during the early diagenetic stage (shallow burial to early cementation stage), while marcasite represents intermediate diagenetic stage. Py II is obviously developed in intermediate to late cementation stage. Microsparite with or without cryptocrystalline quartz usually fill spaces between the iron sulphides. Therefore, they appear to be developed in the late cementation stage which is followed by the formation of the primary diagenetic vugs. The paragenetic sequence of the cementing materials including the iron sulphide and their relation to the developed sedimentary fabrics of the shales is illustrated in Figure 9.

HYPOTHETICAL MODE OF FORMATION

The obtained petrographic observations which are based upon geometric interpretations do suggest the following progressive mechanisms for the diagenetic history of the black shales and the formation of its cementing materials:

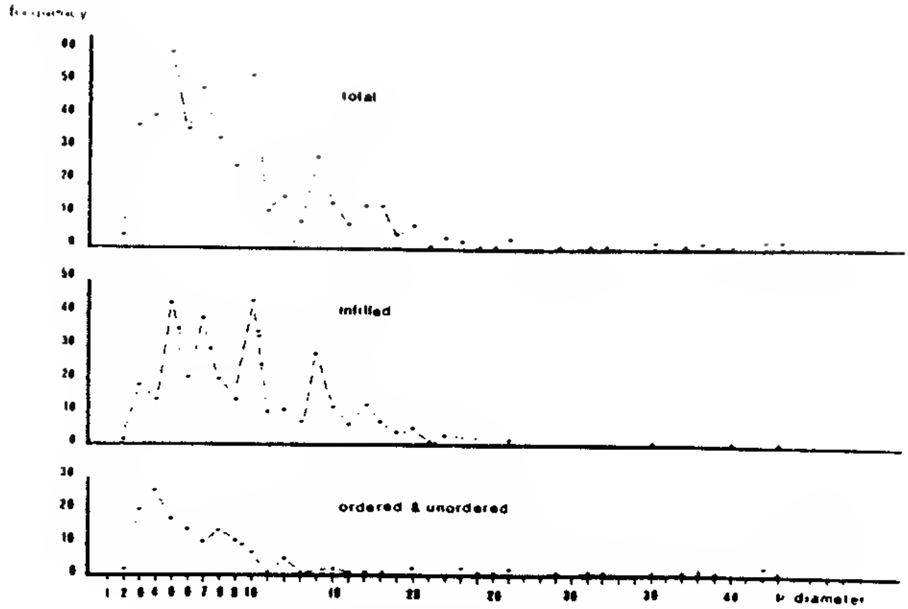

Figure 8. Size distribution of 500 framboids.

1- Burial and rearrangement of accumulated clay fractions and organic skeletal debris under marine calm conditions.

2- Decaying of the soft organic parts of the bioclasts by anaerobic bacteria involving reducing to weakly acidic environment with resulting fossil cavities and empty organic structures.

3- Production of more cyclic and aromatic matter by the concentration of C and H and releasing of S and N (Degen, 1967).

4- Conversion of the clay minerals during the early diagenetic stage to smectite-illite mixed layer and formation of free cations of Mg, Fe, Ca, Na, K with SiO_2 and H_2O (Muller, 1967).

5- Ionic interactions of the free cations and anions in presence of catalytic clay minerals.

6- Fractional crystallization of the cementing materials depending upon microgeochemical changes of the internal physico-chemical parameters of the medium (pH, Eh, pressure, concentration, ..etc.) and producing successive crystallization of pyrite (framboids), marcasite, coarse grained pyrite, calcite and silica, respectively.

7- Maturation of the hydrocarbons giving bitumens and pyrobitumens.

The geometric position and the different grain sizes of the pyrite spherules do indicate that the present framboids are formed during filling processes of the pre-existed fossil or organic cavities. In some instances, they preserve the external and(or) internal micro-structures. The internal geometric characters of framboids prove that they are developed by crystallization of minute pyrite crystals (cubes or pyritohedra) which are usually arranged in closed packing types of spherical outlines. The external spherical forms of the framboids may be developed due to the internal physical character of the pyrite crystals (equidimensional), which causes the spherical form to resist the maximum hydrostatic and lithostatic pressure for the least volume. Accordingly, the present framboids are believed to be formed by primary crystallization and arrangement under distinct physical conditions and as a result of indirect organic origin.

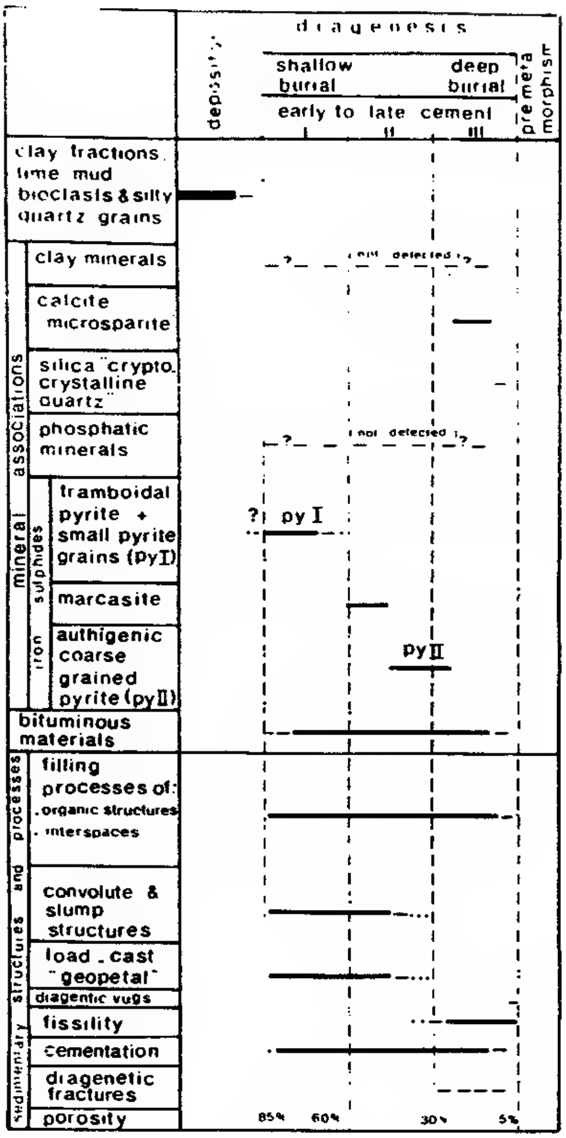

Figure 9. Schematic paragenetic sequence of the iron sulphides during the diagenesis in the black shales.

SUMMARY AND CONCLUSION

1- The Upper Cretaceous black shales of the Duwi Formation contain calcareous foraminiferal tests, phosphatized skeletal nektonic debris, algal and fungal remains, as well as noticeable bituminous matter and considerable amount of iron sulphide.

2- The microlitho- and microbio-facies associations of these rocks suggest that they were deposited in an outer neritic to marginal bathyal zone under deep and calm marine conditons.

3- The iron sulphide occurs in these shales in three different forms : framboidal pyrite with small pyrite crystals, marcasite and coarse grained idiomorphic pyrite.

4- The different iron sulphide forms show a high degree of symmetry and congruency with the sedimentary and syndiagenetic deformational structures of the shale and are closely related to the microorganic structures of the bioclasts. They seem to be developed by filling processes during the diagenesis and before the main consolidation of the country rock.

5- The geometric interrelationships of these iron sulphide forms and the other cementing materials do indicate that they are formed in three successive generations of crystallization or segregation processes corresponding to diagenetic crystallization differentiation. The first crystallizaion generation of the iron sulphide is displayed by the formation of pyrite framboids with small pyrite crystals developing during the early diagenetic stage (shallow burial to early cement). Marcasite displays an intermediate diagenetic stage. However, coarse grained idiomorphic pyrite is obviously developed in intermediate to late diagenetic stage and form the last iron sulphide generation in the crystallization sequence. Calcite and microcrystalline quartz appear to be formed in the late diagenetic stage which followed the formation of the primary voids. The development of these iron sulphide forms appears to be an integral element in the diagenetic history of the studied black shales.

6- Biogenic and physico-chemical conditions are suggested to control the diagenetic differentiations of the different generations of the iron sulphide and the other cementing materials.

7- The pyrite framboids, which constitute a major part of the iron sulphide, are supposed to be developed by primary crystallization and arrangement of minute pyrite crystals under distinct physical conditions and as a result of indirect organic origin.

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عمليات مابعد الترسيب الخاصة بكبريتيدات الحديد الموجودة فى صخور الطفلة السوداء التابعة لعصر الطباشيرى العلوى بالنطاق الساحلى للبحر الأحمر ، مصر

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تحتوى صخور الطفلة السوداء التابعة للعصر الطباشيرى العلوى لمكون الضوى على صدفات من منخريات جيرية وبقايا فقاريات فوسفاتية وبقايا طحالب وفطريات ومواد بتيومينية وكذلك كمية من كبريتيدات الحديد (بيريت وماركازيت) . أثبتت دراسات العلاقات الهندسية بين كبريتيدات الحديد والصخور الحاوية أن هذه الكبريتيدات تظهر درجة عالية من التزامن فى التكوين مع النسيج الرسوبى الأولى للطفلة السوداء وكذلك علاقة مباشرة مع التركيبات العضوية الدقيقة للفتات العضوى .

وقد اتضح أن كبريتيدات الحديد قد تكونت خلال ثلاث مراحل من مراحل مابعد الترسيب . المرحلة الأولى والمتوافقة مع الدفن الضحل تتصف بتكوين البيريت الفرامبويدالى وبعض البللورات الصغيرة من البيريت ، والمرحلة الثانية تتصف بتكوين بللورات الماركازيت وهى متزامنة مع المرحلة المتوسطة من التلاحم ، والمرحلة الثالثة تتصف بتكوين بيريت ذو درجة تبلور عالية وهى التى تتوافق مع المراحل النهائية من التلاحم أو الدفن العميق . تم تصنيف البيريت الفرامبويدالى تبعا لأشكاله الخارجية والبناء الهندسى الداخلى وكذلك درجة تشوهه نتيجة عمليات الملء باضافة بللورات جديدة من البيريت .

ولقد وجد أن الفصل التفاضلى للاشكال المختلفة لكبريتيدات الحديد اثناء عملية مابعد الترسيب قد نتج من تدخل العمليات الحيوية والفيزيقوكيميائية التى من المرجح أن تكون قد حدثت فى بيئة هادئة وعميقة .